

THE OIL OF MAIZE

(ZEA MAYS)

BY

HARRIET WINFIELD, A.M.

SUBMITTED IN PARTIAL FULFILMENT OF THE REQUIRE-
MENTS FOR THE DEGREE OF DOCTOR OF PHILOS-
OPHY IN THE FACULTY OF PURE SCIENCE,
COLUMBIA UNIVERSITY.

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INTRODUCTION.

The work reported in this dissertation was undertaken with the idea of investigating the many conflicting statements as to the properties of maize oil and of furnishing a more complete and consistent record of both its physical and chemical constants than has been heretofore obtainable.

This record has been made as complete as possible and covers many points on which little work seems to have been done by other investigators. The results obtained differ in several important particulars from those previously reported and offer ready means for the identification of the oil.

During the course of the investigation, a method, which is believed to be new, was devised for improving the oil for commercial purposes. This method frees the oil from odor and taste and relieves it of its excess of free acid, at the same time rendering the oil more stable and improving its drying qualities.

For much valuable advice and for samples which could not have been obtained elsewhere, I am indebted to Dr. H. T. Vulté, under whose supervision this work has been conducted.

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THE OIL OF MAIZE.

PART I.—DESCRIPTIVE.

OCCURRENCE AND MANUFACTURE.

The kernel of maize, or Indian corn as it is generally known in this country, contains a larger percentage of oil than does any other grain, with the exception of hulled oats. It contains, in round numbers, twice as much fat as wheat, three times as much as rye and twice as much as barley.¹

The per cent. of fat in the whole kernel varies considerably with season and locality, but is always greater in sweet than in field corn. The following table, compiled by the U. S. Dept. of Agriculture, shows the average composition of American maize.¹

	Field corn.	Sweet corn.
Water	10.75	8.44
Proteids	10.00	11.48
Oil	4.25	8.57
Crude fibre	1.75	2.82
Other carbohydrates	71.75	66.72
Ash	1.50	1.97

While the whole kernel thus shows an unusually large amount of oil, the percentage is much increased if the germ alone be taken into account. The germ of Indian corn is a white, flattened, hornlike body, much lighter than the rest of the kernel and can be pulverized only with difficulty. About 14-16 per cent. of the whole grain is contained in the germ and in this germ the amount of fat varies from 11 per cent. to 22 per cent.

The oil of maize was first known commercially as a bye-product of distilleries. The grain was malted and mashed and the oil was then found floating on the surface of the mash-tubs. The day before the whiskey was distilled, this oil was ladled off and was then purified by washing, filtering, and settling. The oil thus obtained was golden-brown in color, and was so modified in composition by impurities due to its mode of preparation, as

¹ U. S. Dept. Agriculture, Div. of Chem., Bull. 50, 13.

to be practically a distinct oil. This will be seen by a comparison of its constants with those of the normal oil.

The oil was thought to affect injuriously the quality of the whiskey obtained, and various methods were devised to effect its removal before mashing the grain. Of these methods, the most important was that originated by John Crawford of Cincinnati.¹ He extracted the oil from the ground grain by the use of CS_2 and introduced his process into several cities of the United States. He claimed to obtain a larger quantity and a higher grade of whiskey in this way, but his venture was not a commercial success.

A method used for the recovery of the oil from the residues of starch factories is also worthy of note. After the deposition of the starch from the mixture of pulverized corn and water, a milky, easily putrefiable liquid is left, which is an emulsion of oil in the dissolved albuminoids. A method to separate and recover both products was devised by Dr. F. V. Greene, and patented March 10, 1885. Later, the John Scott Legacy Medal of the Franklin Institute was awarded him for his discovery.

The foregoing methods have been almost wholly superseded by the one about to be described, which is practically the only method for the preparation of maize oil in use at the present time. The oil is now an important bye-product in distilleries and also in glucose and in starch factories. In all these manufactures, the grain is first freed from the oil-bearing germ by Mowery's Patent Degerminizer. This machine separates the hard, light germ, first from the crushed grain, and next from the bran or husk, by a process of sifting and winnowing. The separated germ is then steamed to soften it and the oil removed by hydraulic pressure. The oil thus obtained is purified only by straining and settling, no clarifying or bleaching agents being employed for the ordinary commercial product. The residue, or oil cake, is ground and used for cattle feed. It is very valuable for this purpose and is exported in large quantities.

The manufacture of maize oil is assuming larger proportions yearly and is capable of almost indefinite extension.

GENERAL CHARACTERISTICS.

Maize oil, when freshly made, is of a pale straw-yellow color,

¹ Drug. Circ., 1888, 32, 209; J. U. Lloyd: Maize Oil.

and has a strongly-marked, penetrating odor, similar to that of newly ground corn-meal. The color of the oil is probably due to some oxidation, as it is much paler when first made than after standing for some time and develops a bright, golden-yellow with age. The oil recovered from the mash of distilleries is of a pronounced golden-brown color, due to its mode of preparation.

The taste of the oil is at first bland and pleasant, but is followed by considerable acidity, lingering long on the tongue. This taste is as marked in an oil ten years old as in that freshly made.

A considerable variance of opinion as to odor and taste is shown by different writers on the subject of maize oil, but perhaps the most striking judgment is that given by Bizio,¹ the first analyst to study the oil, who, in 1823, described it as of a "vanilla-like odor and balsamic taste."

USES.

Owing to the almost unlimited possible supply of maize oil, and to the cheapness with which it can be produced, much interest attaches to a consideration of the uses for which it is fitted. Unfortunately, the oil has been known primarily as an adulterant of more costly oils and, for this reason, has been seldom sold under its own name in this country.

The most important commercial application of maize oil is its use in the manufacture of soap. It is peculiarly fitted for this purpose, saponifying with great ease in the presence of either hot or cold alkali and forming a light-colored soap of excellent quality. Large quantities of the oil are exported yearly for the use of the English and Belgian soap-makers, but little attention is paid to it for this purpose in America.

Many attempts have been made to utilize maize oil in the manufacture of paint but, owing to its lack of drying properties, most of these attempts have failed to produce anything but a cheap grade of paint. In 1887, however, Geo. W. Banker succeeded in manufacturing a corn-oil paint which, with the aid of suitable dryers, gave fairly satisfactory results. Letters patent were granted him in the year named giving him exclusive rights, throughout Great Britain, Canada, and the United States, in the

¹ Chem. and Phys., 37, 377.

use of maize oil as a vehicle for paints, either alone or in combination with other oils. The specific claims made by Mr. Banker on behalf of maize oil, are as follows:¹

1. White paints do not yellow with age but remain white.
2. Tints, especially those used for indoor work, are more brilliant and durable than when made up with linseed oil.
3. The paint is more homogeneous, not separating into two portions as when linseed oil is used.
4. The paint is smoother in application than a linseed oil paint and possesses greater elasticity.

Considerable attention is now being paid to maize oil by the grinders of paints and its use in this direction may be expected to assume larger proportions.

A third use for which this oil is particularly well fitted is as a substitute for olive oil in the Pharmacopoeia. In nearly all preparations in which a fixed oil is necessary, the oil of maize is equal or superior to any other available oil, and its use should be made official. This is notably true in the case of ointments, such as camphor cerate, and of ammoniacal and lead liniments.²

Notwithstanding the percentage of free acid in maize oil, and the marked action which it has on metal, an effort has been made to introduce it as a lubricant for machinery. Three patents were issued to Geo. W. Banker,³ in the years 1882-1884, granting him exclusive rights for the combination of :

1. Corn oil, castor oil and petroleum.
2. Corn oil with mineral oil, for lubricating and other purposes.
3. Corn oil and castor oil.

As castor oil may be made to combine with any gravity of paraffin, or of crude or refined petroleum, by the use of corn oil, any grade of lubricating oil may be made in this way.

Maize oil has also been used to some extent as an illuminant, burning with a white flame and evolving quite a high degree of heat. Among other uses which may be named for this oil are :

1. An adulterant for linseed and other paint oils.
2. An adulterant for lard.
3. An adulterant for olive and other salad oils.
4. A vehicle for the introduction of coloring-matter into butter.
5. A substitute for linseed oil in the manufacture of putty.

¹ Oil, Paint and Drug Reporter, Oct. 12, 1891.

² Heinitsh : Pharm. Rec., 1889, 9, 236; Lloyd : Drug. Circ., 1888, 32, 209.

³ Oil, Paint and Drug Reporter, Oct. 12, 1891.

PART II.—ANALYTICAL.

CONSTITUTION.

Although the actual separation and determination of the mixed fatty acids contained in maize oil have not been attempted in the present investigation, certain inferences as to the constitution of the oil have resulted from observations made during the progress of the work. These inferences, together with a brief résumé of the work done in this line by other analysts, may be found of some interest at this point.

The presence of a volatile oil, with a "peculiar grain-like odor," has been recognized by nearly all observers. This volatile oil is very characteristic of the oil of maize, but may be easily and completely driven off by a steam distillation at the ordinary pressure, leaving the oil free from all objectionable odor and taste.

In the year 1866, Hoppe-Seyler¹ succeeded in isolating stearic, palmitic, and oleic acids from maize oil and the existence of these acids has since been confirmed by many observers. Rokiti-ansky,² in 1894, added linolic acid to those previously determined and succeeded in obtaining its oxidation product, sativic or tetraoxystearic acid. The presence of linolic acid is confirmed by a late observer, C. G. Hopkins,³ of Cornell University, who finds a large percentage of this acid in maize oil.

Rokitiansky¹ also asserts the presence of a hydroxylated acid (probably ricinoleic) in the solid fatty acids, and this result is confirmed by the somewhat high acetyl figure of the oil. The evidence of the existence of linolenic acid is thus far negative and the only insoluble fatty acids, whose presence in maize oil can be affirmed with any degree of certainty are :

1. Palmitic acid = $C_{16}H_{32}O_2 = C_{15}H_{31}COOH$.
2. Stearic acid = $C_{18}H_{36}O_2 = C_{17}H_{35}COOH$.
3. Oleic acid = $C_{18}H_{34}O_2 = C_{17}H_{33}COOH$.
4. Linolic acid = $C_{18}H_{32}O_2 = C_{17}H_{31}COOH$.
5. Ricinoleic acid = $C_{18}H_{34}O_3 = C_{17}H_{32}(OH)COOH$.

Comparatively little attention has been paid to the volatile acids of maize oil, certain observers going so far as to deny their

¹ Bull. Soc. Chim. (1866), [2] 6, 342.

² Ph. Russ. (1894), 712-713.

³ J. Am. Chem. Soc., Dec., 1898.

existence. Rokitiānsky,¹ however, has succeeded in obtaining proof of the presence of formic acid, and also asserts that caproic, caprylic, and capric acids are probably to be found in the oil. A steam distillation of the mixed fatty acids, during the course of the present investigation, disclosed the presence of a notable amount of some acid, volatile in steam, but insoluble in water. This is probably lauric acid.

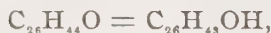
The following points are indications that homologues of the acetic series, lower than lauric acid, are to be found in maize oil :

1. The high Reichert value.
2. The high percentage of glycerine.
3. The ease with which partial dissociation of the oil takes place under the influence of steam at the ordinary pressure.
4. The fact that the aqueous liquid, obtained by washing the mixed fatty acids with boiling water and decanting through a filter, shows a marked acid reaction to phenolphthalein, after having been made neutral to methyl orange.

The probable presence of the following volatile acids in maize oil may, therefore, be affirmed. Much further investigation, however, is necessary to establish its constitution, both in regard to the soluble and to the insoluble fatty acids.

1. Formic acid = $\text{CH}_2\text{O}_2 = \text{HCOOH}$.
2. Caproic acid = $\text{C}_6\text{H}_{12}\text{O}_2 = \text{C}_6\text{H}_{21}\text{COOH}$.
3. Caprylic acid = $\text{C}_8\text{H}_{16}\text{O}_2 = \text{C}_7\text{H}_{15}\text{COOH}$.
4. Capric acid = $\text{C}_{10}\text{H}_{20}\text{O}_2 = \text{C}_9\text{H}_{19}\text{COOH}$.
5. Lauric acid = $\text{C}_{12}\text{H}_{24}\text{O}_2 = \text{C}_{11}\text{H}_{23}\text{COOH}$.

The unsaponifiable matter consists almost wholly of phytosterol or phytosteryl alcohol,



and the amount contained is very characteristic of the oil.

DETERMINATION OF SPECIFIC GRAVITY.

Description of Method.

A. Sprengel Tube.—The tubes used in this determination were made in the usual shape, from small glass tubing and supplied with a platinum wire by which to suspend them from the balance hook.

¹ Ph. Russ. (1894), 712-713.

Each tube was weighed, filled with distilled water at 15.5° C. and again weighed. It was then carefully dried, cooled, filled with the oil at 15.5° C. and again weighed, thus affording data for the determination of the specific gravity at 15.5° C.

In order to obtain the specific gravity at 100° C., the Sprengel tube, filled with oil, was placed in the neck of an Erlenmeyer flask, in such a manner that the capillary arms of the tube were supported by the neck of the flask. The tube was then subjected to the action of boiling water until oily drops ceased to ooze from the capillaries, when it was again dried, cooled and weighed. The oily drops were removed as they appeared by a piece of filter-paper. The weight of the oil at 100° C. was compared with that of distilled water at 15.5° C.

B. Westphal Balance.—This method, although it can be quickly applied, does not compare in accuracy with that by means of the Sprengel tube. In fact, very little reliance can be placed on it beyond the second place of decimals.

In obtaining the specific gravity at 100° C. by this method, a solid thermometer body was used, and the tube containing the oil was immersed in boiling water, no attempt being made to take the gravity until the oil was uniformly at 100° C. Still more variation was observed when using the Westphal at this temperature than at 15.5° C.

RESULTS OBTAINED.

A. PHOENIX PAINT CO. OIL.¹

	Wt. water 15.5° C. Grams.	Wt. oil 15.5° C. Grams.	Wt. oil 100° C. Grams.	Sp. gr. 15.5° C.	Sp. gr. 100° C.
I	1.5265	1.4068	1.3315	0.9216	0.8722
II	1.6404	1.5113	1.4297	0.9213	0.8715
III	1.3602	1.2528	1.1848	0.9210	0.8710
Average for Sprengel tube.....				0.9213	0.8716
Westphal balance.....				0.921	0.895

B. CHICAGO GLUCOSE SUGAR REFINING CO. OIL.²

I	2.0130	1.8547	1.7554	0.92136	0.87203
II	2.1788	2.0072	1.8958	0.92124	0.87011
Average for Sprengel tube.....				0.9213	0.87107
Westphal balance.....				0.921	

¹ Ten-year-old sample of oil expressed by hydraulic pressure.

² Fresh sample of oil expressed by hydraulic pressure.

C. DISTILLERY OIL.¹

	Wt. water 15.5° C. Grams.	Wt. oil 15.5° C. Grams.	Wt. oil 100° C. Grams.	Sp. gr. 15.5° C.	Sp. gr. 100° C.
I	1.0037	0.9288	0.8789	0.9253	0.8746
II	1.5718	1.4551	1.3780	0.9257	0.8767
Average for Sprengel tube.....				0.9255	0.8756
Westphal balance.....				0.9255	

COMPARISON WITH RESULTS OF OTHER OBSERVERS.

Sp. gr. at 15° C.	Observer.	Reference.
0.8360	Rokitiński ²	Ph. Russ. (1894), 712-713.
0.9160	Curtmann	Chem. Centrbl., 59, 1193.
0.9170	Bowers ³	Pharm. J., Nov., 1889.
0.9200	Shuttleworth	Pharm. J., 16, 1095.
0.9215	Schaedler	J. Soc. Chem. Ind., 11, 504.
0.9216	Procter	J. Soc. Chem. Ind., 17, 11.
0.9215-0.9244	De Negri and Fabris	Ztschr. anal. Chem., 33, 547-72.
0.9220	Trimble	Am. J. Pharm., 58, 265.
0.9239	Hart ³	Chem. Ztg., 17, 1522.
0.9238-0.9262	Hopkins	J. Am. Chem. Soc., Dec., 1898.
0.9243	Dulière	J. Pharm. (1897), 217.
0.9244	Smith	J. Soc. Chem. Ind., 11, 504-5.
0.9245	DeNegri ²	Chem. Ztg., 22, 961-976.
0.9262	Mills	J. Soc. Chem. Ind., 11, 504-5.

DETERMINATION OF VISCOSITY.

Process.

This determination was made by means of a Boverton Redwood viscosimeter. The viscosity was taken at 20° C. and the instrument carefully standardized for both distilled water and rape oil at this temperature. The time of flow for 50 cc. oil was noted by means of a stop-watch and six readings were made for each sample. Great care was taken to avoid any change of temperature during the operation.

Specific Viscosity.

Distilled Water as Standard.—Time of flow for 50 cc. oil as compared with that of equal volume of distilled water at the same temperature.

Rape Oil as Standard.—Viscosity determined in manner similar to that used with water-standard, but a correction applied by multiplying the result by the ratio of the density of the oil under examination to that of rape oil. Viscosity of rape oil taken as 100.

¹ Fifteen-year-old sample of oil from mash of distillery.

² Petroleum ether extract.

³ Dark brown oil.

RESULTS OBTAINED.

	Tempera- ture.	Av. time of flow. Seconds.	Viscos. water.	Viscos. rape oil.
Distilled water	20°	29	1.00	
Rape oil	20°	405.5		100.00
Phoenix oil	20°	283.7	9.79	70.42
Glucose sugar oil	20°	297.7	10.27	73.89

COMPARISON WITH RESULTS OF OTHER OBSERVERS.

Tempera- ture.	Viscos. water.	Viscos. rape.	Observer.	Reference.
18° C.-19° C.		61.1	Smith	J. Soc. Chem. Ind., 11, 504.
15° C.	19.2		Andés	Veg. Fats and Oils.
Viscosity of almond oil			Shuttleworth	Pharm. J., 16, 1095.
Viscosity greater than olive			Bowers	Pharm. J., Nov., 1889.

DETERMINATION OF INDEX OF REFRACTION.

The instrument used for this determination was Abbé's Refractometer.

A. PHOENIX PAINT CO. OIL.

	Temperature.	A.	T.
I.....	15° C.	1.4768	35.8
II.....	15° C.	1.4766	36.0
III.....	20° C.	1.4762	35.9
IV.....	20° C.	1.4760	35.9
Average for.....	15° C.	1.4767	35.9
Average for.....	20° C.	1.4761	35.9

B. CHICAGO GLUCOSE SUGAR REFINING CO. OIL.

I.....	15° C.	1.4765	35.8
II.....	15° C.	1.4766	36.0
III.....	15° C.	1.4767	36.0
Average for	15° C.	1.4766	35.9

C. DISTILLERY OIL.

I.....	19.5° C.	1.4767	36.2
II.....	20° C.	1.4766	36.2
III.....	20.5° C.	1.4764	36.3
Average for	20° C.	1.4765	36.2

COMPARISON WITH RESULTS OF OTHER OBSERVERS.

Tempera- ture.	Refractive index.	Observer.	Reference.
15° C.	1.4765	Procter	J. Soc. Chem. Ind., 17, 11.

DETERMINATION OF ASH.

Process.

The oil was weighed into a small, flat-bottomed platinum dish, and the operation conducted in the usual manner, care being taken to employ as low a temperature as possible. The odor of the volatile oil was very marked at first but soon entirely disappeared. The oil burned with a very bright flame and comparatively little smoke.

RESULTS OBTAINED.

A. PHOENIX PAINT CO. OIL.

	Wt. oil. Grams.	Wt. ash. Gram.	Ash. Per cent.
I	2.9346	0.0019	0.064
II	2.2665	0.0015	0.066
Average			0.065

B. DISTILLERY OIL.

I	0.6750	0.0011	0.065
II	0.6005	0.0004	0.066
Average			0.0655

DETERMINATION OF FREE ACID.

Process.

Five to ten grams oil were carefully weighed into a 4-oz. flask. 50-100 cc. of a mixture of 9 parts 95 per cent. alcohol and 1 part ether were neutralized with aqueous $n/10$ KOH, phenolphthalein indicator, and added to contents of flask. The flask was then heated on a water-bath until the solution of the oil was as nearly perfect as possible, and the liquid in the flask was finally titrated, while still warm, with $n/10$ KOH.

Acid Value.

Number mg. KOH necessary to neutralize 1 gram oil.

Per Cent. Free Acid.

This constant was calculated to oleic acid.

Degrees of Acidity.

Number cc. n /KOH required to neutralize 100 grams oil.

Action on Copper.

Bright copper wire was immersed in the oil and allowed to remain for several days. Marked discoloration was shown by all three oils, the glucose sugar oil being the least affected.

RESULTS OBTAINED.

A. PHOENIX PAINT CO. OIL.

	Wt. oil. Grams.	Vol. $n/10$ KOH. cc.	Acid value.	Free acid. Per cent.	Degrees acidity.
I	10.6617	7.0	3.68	1.851	6.56
II	7.2552	4.8	3.71	1.851	6.62
Average.....			3.70	1.851	6.59

B. CHICAGO GLUCOSE SUGAR REFINING CO. OIL.

I	9.9294	4.0	2.26	1.136	4.03
II	13.0816	5.2	2.23	1.121	3.97
Average.....			2.25	1.128	4.00

C. DISTILLERY OIL.

I	4.2351	15.6	20.64	10.387	36.83
II	6.2726	23.1	20.66	10.385	36.83
Average.....			20.65	10.386	36.83

COMPARISON WITH RESULTS OF OTHER OBSERVERS.

Per cent. free acid.	Observer.	Reference.
0.75	Hart ²	Chem. Ztg., 17, 1522.
0.88	Lloyd	Chem. Centrbl., 59, 257.
0.00	Spüller ³	Dingl., 264, 626.
5.65	De Negri ¹	Chem. Ztg., 22, 961-976.

DETERMINATION OF IODINE ABSORPTION.

*Description of Method.**A. Necessary Solutions.*

1. HgCl_2 in 95 per cent. alcohol = 60 grams per liter.
2. Iodine in 95 per cent. alcohol = 50 grams per liter.

The alcohol used in making up these solutions was purified by oxidation with $\text{K}_2\text{Mn}_2\text{O}_8$ and subsequent distillation over pulverized CaCO_3 . The distillate was rejected until no discoloration from aldehyde-resin was shown on heating a small portion with a lump of solid KOH for ten minutes in boiling water..

3. $n/10 \text{ Na}_2\text{S}_2\text{O}_3$.

This solution was standardized by $n/50 \text{ K}_2\text{Cr}_2\text{O}_7$ and proved to contain 24.3137 grams of $\text{Na}_2\text{S}_2\text{O}_3$.

4. Twenty per cent. KI solution made up with distilled water.
5. Solution of boiled starch as indicator.

*B. Process.*¹ Petroleum ether extract.² Dark-brown oil.³ Ether extract.

Thin Erlenmeyer flasks, having accurately fitted ground glass stoppers and flaring mouths, thus forming a gutter between flask and stopper, were used for this operation.

Equal parts of solutions 1 and 2 were mixed 24 hours before each test and allowed to stand in the dark until needed.

About 0.250 gram oil was weighed into each flask, the oil dissolved in 10 cc. chloroform and 25 cc. of the mixed Hübl solution added. The flask was then stoppered, the gutter filled with KI solution and the whole set away in the dark for 24 hours. A blank test was run for every determination.

After 24 hours the stopper was removed and distilled water added to contents of flask until of convenient bulk for titration. KI solution was also added, sufficient to dissolve any precipitated HgI_2 . The liquid was then titrated with $n/10$ $\text{Na}_2\text{S}_2\text{O}_3$ until colorless, a few drops of starch indicator being added when color had nearly disappeared.

RESULTS OBTAINED.

A. PHOENIX PAINT CO. OIL.

	Wt. oil. Gram.	Vol. hypo. cc.	Wt. iodine. Gram.	Iodine absorption. Per cent.
I.....	0.1986	18.9	0.235323	118.44
II.....	0.2466	23.7	0.295088	119.66
III.....	0.2677	26.0	0.323725	120.90
IV.....	0.22835	22.0	0.273921	119.95
Average Hübl figure.....				119.74

B. CHICAGO GLUCOSE SUGAR REFINING CO. OIL.

I.....	0.2708	26.2	0.322183	118.97
II.....	0.2441	23.3	0.290108	118.85
III.....	0.2697	25.7	0.319990	118.65
IV.....	0.3070	29.1	0.326232	118.02
Average Hübl figure.....				118.62

C. DISTILLERY OIL.

I.....	0.2199	20.1	0.250264	113.80
II.....	0.2894	26.2	0.326215	112.72
III.....	0.2559	23.4	0.291352	113.85
IV.....	0.2453	22.2	0.276471	112.70
Average Hübl figure.....				113.27

COMPARISON WITH RESULTS OF OTHER OBSERVERS.

Hübl figure.	Observer.	Reference.
75.8	Rokitianski ¹	Ph. Russ. (1894), 712-713.
111.2-123	De Negri and Fabris	Ztschr. anal. Chem., 33, 547-72.
115.17	De Negri ²	Chem. Ztg., 22, 961-976.
116.3	Smetham	Anal., 18, 191-193.
117	Hart ²	Chem. Ztg., 17, 1522.
119.6	Hazura	Ztschr. angew. Chem. (1888), 696.
119.4-119.9	Spüller ³	Dingl., 264, 626.
121.7-122.7	Lane	J. Chem. Soc. (1893), A, 153.
122	Hehner	J. Soc. Chem. Ind., 16, 87.
122	Wallenstein	Chem. Ztg. (1894), 18, (ii), 119.
121.5-123.1	Hopkins	J. Am. Chem. Soc., Dec., 1898.
122.55	Dulière	J. Pharm. (1897), 217.
122.9	Mills	J. Soc. Chem. Ind., 11, 504.

DETERMINATION OF SAPONIFICATION VALUE.

Koettstorfer Process.

About 2.5 grams of oil were weighed into a 4-oz. flask and 25 cc. alcoholic potash, approximately half-normal, and made from alcohol purified as described under Hübl process, were added. A blank test was also run on an equal amount of alcoholic potash in a similar flask. Blank and test were covered with watch-glasses, to avoid contamination by CO₂, and evaporated to complete dryness on a water-bath. 75 cc. neutral alcohol were next added to the contents of each flask, heat applied until solution was perfect and liquid titrated, while still warm, until n/HCl , phenolphthalein indicator. The blank was titrated first, on account of its greater liability to contamination by CO₂.

Saponification Value or Koettstorfer Figure.

Number of mg. of KOH necessary for saponification of one gram of oil.

Saponification Equivalent.

Number of mg. of oil equivalent to 1 cc. n/KOH .

¹ Petroleum ether extract.

² Dark brown oil.

³ Ether extract.

RESULTS OBTAINED.

A. PHOENIX PAINT CO. OIL.

	Wt. oil. Grams.	Vol. n/HCl. cc.	Koettstorfer fig.	Sapon. equiv.	Ether value.
I	2.0127	6.95	193.71	289.61	190.01
II	2.5187	8.60	191.55	292.88	187.85
III	2.4600	8.45	192.70	291.13	189.00
Average.....			192.65	291.21	188.95

B. CHICAGO GLUCOSE SUGAR REFINING CO. OIL.

I	3.8356	13.20	193.07	290.57	190.82
II	5.2317	18.05	193.55	289.85	191.30
III	2.5611	8.75	191.31	293.24	189.06
Average.....			192.64	291.22	190.39

C. DISTILLERY OIL.

I	1.9700	6.75	190.29	294.81	169.64
II	2.2206	7.65	193.26	290.28	172.61
Average.....			191.78	292.55	171.13

COMPARISON WITH RESULTS OF OTHER OBSERVERS.

Koettstorfer fig.	Sapon. equiv.	Observer.	Reference.
182.81	306.9	De Negri ¹	Chem. Ztg., 22, 961-976.
188.1-189.2	298.3-296.6	Spüller ³	Dingl., 264, 626.
188-193	298.4-290.7	De Negri and Fabris	Ztschr. anal. Chem., 33, 547.
189.5	296	Hart ²	Chem. Ztg., 17, 1522.
193.4	290.07	Mills	J. Soc. Chem. Ind., 11, 504.
198.5	282.6	Smetham	Anal. 18, 191-193.
198.8-203	282.2-276.4	Dulière	J. Pharm. (1897), 217.

DETERMINATION OF INSOLUBLE FATTY ACIDS.

Hehner and Angell's "Wash Process."

2-4 grams oil were dissolved in ether in a beaker. 50 cc. of alcoholic potash (made by dissolving about 20 grams KOH in 500 cc. purified and redistilled 95 per cent. alcohol), were added to ether solution of oil and the whole heated on a water-bath until saponification was effected. The liquid was then diluted with hot distilled water and heated until the ether and alcohol were entirely expelled. The aqueous soap solution thus formed was broken up with dilute HCl and heating continued until insoluble fatty acids formed a clear oily layer.

¹ Petroleum ether extract.² Dark brown oil.³ Ether extract.

The liquid was then chilled until fatty acids were solidified and the watery portion decanted through an ashless filter. Boiling water was added to fatty acids remaining in beaker and chilling and decanting repeated. Process was continued until filtrate was neutral to methyl orange. Fatty acids were finally transferred to filter and allowed to run through into tared flask. Filter was washed out with ether, this expelled from flask in an air-bath and the flask was then cooled and weighed.

RESULTS OBTAINED.

A. PHOENIX PAINT CO. OIL.

	Wt. oil taken. Grams.	Wt. insol. fatty acids. Grams.	Hehner. value.
I.....	4.6700	4.3256	92.63
II.....	2.6092	2.4121	92.45
III.....	2.3161	2.1609	93.29
Average Hehner value.....			92.79

B. CHICAGO GLUCOSE SUGAR REFINING CO. OIL.

I.....	3.5853	3.2678	91.14
II.....	3.2331	3.0142	93.23
III.....	2.6079	2.4079	92.33
Average Hehner value.....			92.23

C. DISTILLERY OIL.

I.....	3.9832	3.5102	88.12
II.....	2.8297	2.4995	88.30
Average Hehner value.....			88.21

COMPARISON WITH RESULTS OF OTHER OBSERVERS.

Hehner value.	Observer.	Reference.
93.40	Hoppe-Seyler ²	Bull. Soc. chim. (1866), [2], 6, 342.
93.57	Hopkins	J. Am. Chem. Soc., Dec., 1898.
94.70	Spüller ²	Dingl., 264, 626.
95.70	Hart ¹	Chim. Ztg., 17, 1522.
96.70	Lloyd	Chem. Centrbl., 59, 1193.

DETERMINATION OF VOLATILE ACIDS.

Reichert (Modified) Process.

Precisely 2.5 grams oil were weighed into a small wide-mouthed flask and saponified by 25 cc. of approximately half-normal alcoholic potash, as in the Koettstorfer process, evaporating off the alcohol completely. The alcohol used in this determination was

¹ Dark brown oil.² Ether extract.

purified as described under the Hübl process. 50 cc. distilled water, containing 1 cc. phenolphthalein indicator, were then added to the dried soap and the whole heated on the water-bath until the soap was completely dissolved. While still warm, the aqueous soap solution was titrated with $n/2$ H_2SO_4 , overrunning 2 cc. The decomposed soap solution was then slowly distilled into a similar flask, containing 50 cc. of $n/10$ KOH plus 1 cc. phenolphthalein indicator. Great care was used to prevent any of the liquid being carried over into the receiver. A large percentage of a solid fatty acid also distilled over in white flakes, but was held back by a small, wetted filter, placed in the neck of the receiving flask.

When about 50 cc. of the liquid in the distilling flask had gone over, 50 cc. more of distilled water were added to the residue and the distillation repeated. The contents of the receiver were then titrated back with $n/10$ HCl and the "Reichert Figure" calculated from the amount of volatile acids thus recovered from the two distillations.

Reichert Figure.

Number of cc.'s of $n/10$ KOH neutralized by the volatile acids recovered from two distillations of the fatty acids from 2.5 grams oil.

RESULTS OBTAINED.

A. PHOENIX PAINT CO. OIL.

	Vol. $n/10$ HCl.	Reichert figure.	Wt. KOH for 100 g. oil. Gram.
I	45.8	4.2	0.94248
II.....	45.6	4.4	0.98736
Average.....		4.3	0.96492

R. GLUCOSE SUGAR REFINING CO. OIL.

I	46.0	4.0	0.89760
II.....	45.6	4.4	0.98736
Average.....		4.2	0.94248

COMPARISON WITH THE RESULTS OF OTHER OBSERVERS.

Reichert value.	Observer.	Reference.
0.33	Spüller	Dingl., 264, 626.
2.5 ¹	Smith	J. Soc. Chem. Ind., 11, 504.
6-7 ²	Morse	N. H. Expt. Sta. Bull. (1892), 16, 19.
0.0	Hopkins	J. Am. Chem. Soc., Dec., 1898.

¹ Calculated. 100 parts oil require 0.56 parts KOH.

² Calculated. Reported as 3.2 per cent. volatile acids.

C. DISTILLERY OIL.

	Vol. $n/_{10}$ HCl.	Reichert figure.	Wt. KOH for 100 g. oil. Grams.
I	39.9	10.1	2.26644
II.....	40.3	9.7	2.17668
Average.....		9.9	2.22156

DETERMINATION OF ACETYL VALUE.

Process.

About 50 grams oil were placed in a round-bottomed flask, together with an equal volume of acetic anhydride, the flask attached to an inverted condenser and its contents slowly boiled for two hours. The acetylated oil was next transferred to a large beaker, several hundred cc. of distilled water added and the liquid heated to the boiling-point for about half an hour. The contents of the beaker were then transferred to a separator, the aqueous layer drawn off, the acetylated oil washed with boiling water until acid-free (methyl orange indicator), filtered through a dry filter and dried in an air-bath at 100° C.

2-4 grams of the dried, acetylated oil were now saponified with alcoholic potash, a blank being run to ascertain the titre of the potash used and care taken not to heat the liquid longer than was necessary for saponification. The Koettstorfer figure was then ascertained in the usual manner by titration with n/HCl . Enough additional n/HCl was then run into the liquid to make the total amount of acid exactly correspond to the titre of the alcoholic potash used. The oily layer was next filtered off and washed until acid-free as in the Hehner process. Finally the filtrate and washings were titrated with n/KOH .

Acetyl Value.

Number of mg. KOH necessary to neutralize acetic acid resulting from saponification of one gram of acetylated oil.

RESULTS OBTAINED.

A. PHOENIX PAINT CO. OIL.

	Wt. oil. Grams.	Vol. n/HCl . cc.	Koettstorfer fig.	Vol. $n/_{10}$ KOH. cc.	Acetyl value.
I.....	2.3420	8.8	210.8	4.5	10.78
II.....	2.0092	7.6	212.2	4.1	11.45
Average.....			211.5	..	11.12

B. CHICAGO GLUCOSE SUGAR REFINING CO. OIL.

	Wt. oil. Grams.	Vol. n/HCl . cc.	Koettstorfer fig.	Vol. $n/10KOH$. cc.	Acetyl value.
I.....	1.8641	7.1	213.7	3.7	11.14
II.....	1.4686	5.5	210.1	3.1	11.84
Average.....			211.9	..	11.49

COMPARISON WITH RESULTS OF OTHER OBSERVERS.

Koetts- torfer fig.	Acetyl value (filtr.).	Observer.	Reference.
201.5	8.25	Lewkowitsch	Allen: Com. Org. Anal., 2, 68.
200.9	7.90	Lewkowitsch	Allen: Com. Org. Anal., 2, 68.

DETERMINATION OF GLYCEROL.

*Hehner's Dichromate Method.**Solutions.*

1. $K_2Cr_2O_7 = 74.86$ grams per liter.
2. $K_2Cr_2O_7 = 7.486$ grams per liter.
3. $FeSO_4 \cdot (NH_4)_2SO_4 \cdot 6H_2O =$ about 240 grams per liter.

Solution 1 was made with the utmost care from $K_2Cr_2O_7$ of known purity. 1 cc. of this solution corresponds to 0.010025 gram of glycerol.

Solution 3 was standardized on solution 2; a little H_2SO_4 was added to the solution to prevent decomposition.

Process.

2-3 grams oil were saponified with alcoholic potash and the soap solution at once diluted to about 200 cc., the alcohol not being driven off on account of danger of loss from volatilization of glycerol. The soap was then decomposed by dilute H_2SO_4 , the liberated fatty acids filtered off and washed until acid-free and the filtrate and washings, amounting in all to about 1 liter, reduced to 250 cc. by evaporation.

50 cc. of the stronger dichromate solution and 25 cc. of concentrated H_2SO_4 , suitably diluted, were next added to the glycerol solution and the whole heated on a water-bath for about two hours. The liquid was then titrated with an excess of the ferrous solution and this excess titrated back with the dilute dichromate solution, potassium ferricyanide indicator.

RESULTS OBTAINED.

A. PHOENIX PAINT CO. OIL.

	Wt. oil. Grams.	Vol. $K_2Cr_2O_7$. cc.	Wt. glycerol. Grams.	Glycerol. Per cent.
I.....	2.6092	27.57	0.276395	10.59
II.....	2.3361	24.46	0.245217	10.50
Average				10.545

B. CHICAGO GLUCOSE SUGAR REFINING CO. OIL.

I.....	2.5701	26.37	0.264359	10.29
II.....	2.0425	21.21	0.212635	10.41
Average				10.35

DETERMINATION OF PHYTOSTEROL.

Process of Foster and Reichelmann.

50 grams oil were weighed into a round-bottomed flask, 75 cc. of 95 per cent. alcohol added and the mixture boiled for 5 minutes under a reflux condenser. While hot, the alcohol was decanted through a filter, and the oil was then treated with a fresh portion of alcohol. The alcoholic filtrate was next saponified with an excess of aqueous potash, the resultant soap evaporated to dryness and the residue extracted with successive portions of ether.

The ether extract was evaporated to dryness and the residue of crude phytosterol estimated as unsaponifiable matter.¹ The residue was then treated with a little ether, the resultant solution filtered and ether evaporated, this second residue taken up with 95 per cent. alcohol and the phytosterol crystallized out. The phytosterol crystallized in well-marked needle-shaped crystals, occurring in tufts.

RESULTS OBTAINED.

	Wt. oil. Grams.	Wt. ether residue. Gram.	Unsap. matter. Per cent.
Phoenix paint oil	55.3945	0.7691	1.39
Glucose sugar oil.....	49.1123	0.7036	1.43
Average			1.41

COMPARISON WITH RESULTS OF OTHER OBSERVERS.

Unsap. matter. Per cent.	Observer.	Reference.
1.35	Spüller	Dingl., 264, 626.
1.55	Hart ²	Chem. Ztg., 17, 1522.
2.86	Hopkins	J. Am. Chem. Soc., Dec., 1898.

¹ Possible traces of hydrocarbon oils neglected by this method.

² Dark brown oil.

COLOR REACTIONS WITH SULPHURIC ACID.

I. Heydenreich's Test.

Process.—20 drops of oil were placed on a watch-glass and two drops of H_2SO_4 , sp. gr. 1.84, allowed to fall into center of oil. The oil and acid were then stirred together with a glass rod.

Results Obtained.

A. Phoenix Paint Oil—Glucose Sugar Oil.—Before stirring, rayed ring of mahogany red on golden-brown background; after stirring, dark red-brown with honey-like consistency.

B. Distillery Oil.—Before stirring, similar to result with the other samples; after stirring, dull claret with honey-like consistency.

II. Carbon Disulphide Test.

Process.—One drop concentrated H_2SO_4 was added to solution of a few drops oil in CS_2 ; the mixture was then well shaken and allowed to stand for 24 hours.

Results Obtained.

A. Phoenix Paint Oil—Glucose Sugar Oil.—Golden-brown color by transmitted light; mahogany-red by reflected light. After 24 hours, fine violet.

B. Distillery Oil.—Deep claret color; after 24 hours, fine violet.

COMPARISON WITH RESULTS OF OTHER OBSERVERS.

HNO_3 .	H_2SO_4 .	Observer.	Reference.
Yellow-orange		Dulière	J. Pharm. (1897), 217.
.....	Dark red	Hart ¹	Chem. Ztg., 17, 1522.
Reddish (Massie)	Black-brown	Shuttleworth	Pharm. J., 16, 1095.
Reddish-yellow	Green	Brann	An. and Veg. Fats and Oils.

COLOR REACTIONS WITH NITRIC ACID.

I. Hauchecorne's Test.

Process.—3–5 parts of oil by volume were shaken with one part HNO_3 , of sp. gr 1.32. Mixture was then heated on water-bath for five minutes and allowed to stand.

Results Obtained.

A. Phoenix Paint Co. Oil—Glucose Sugar Oil.—Oily layer orange yellow and of consistency of thick honey.

¹ Dark brown oil.

B. Distillery Oil.—Oily layer mahogany red and of much less viscosity.

II. Massie's Test.

Process.—3 parts oil by volume were shaken with one part HNO_3 , of sp. gr. 1.42, for two minutes and allowed to stand.

Results Obtained.

A. Phoenix Paint Oil—Glucose Sugar Oil.—Oily layer bright mahogany red, solidifying after some days.

B. Distillery Oil.—Oily layer very dark reddish-brown and of consistency of strained honey.

III. Glassner's Test.

Process.—Oil was cautiously poured into equal volume of fuming HNO_3 , using very small quantities.

Results Obtained.

A. Phoenix Paint Oil—Glucose Sugar Oil.—After much effervescence, oil was changed to pale yellow solid with rancid odor.

B. Distillery Oil.—After much effervescence, oil became of a brilliant orange-yellow and of the consistency of thick honey.

SILVER NITRATE REDUCTION TESTS.

I. Becchi's Test.

Process of Pearmain and Moor.—10 cc. oil were shaken with 2 cc. of a reagent prepared by dissolving 1 gram of AgNO_3 in 100 cc. of 95 per cent. alcohol, adding 20 cc. ether and one drop of nitric acid. The mixture was then placed in boiling water for ten minutes.

Results Obtained.

Phoenix Paint Oil—Glucose Sugar Oil.—Dark brown coloration. Viscosity increased.

Distillery Oil.—Dark brown coloration. Viscosity much increased.

II. Brullé's Test.

Process.—12 cc. oil were shaken with 5 cc. of solution of 2.5 grams AgNO_3 in 100 cc. of 95 per cent. alcohol. The mixture was then heated in boiling water 20 minutes.

Results Obtained.

All three oils colored intensely black.

MISCELLANEOUS COLOR REACTIONS.

I. Wellman's Test.

Process.—2 cc. of freshly made phosphomolybdic acid were shaken with solution of 25 drops oil in 5 cc. chloroform. A few drops of NH_4OH were then added, the mixture again shaken and allowed to separate.

Results Obtained.

A. Phoenix Paint Oil—Glucose Sugar Oil.—Very dark green upper layer, becoming bright blue on addition of NH_4OH .

B. Distillery Oil.—Coloring much lighter than in case of the other two oils.

II. Renard's Test

Process.—10 cc. of reagent, composed of equal parts SnBr_2 and CS_2 , were shaken with 5 cc. oil.

Results Obtained.—Brown-violet coloration, with slight evolution of gas, for all three oils.

III. Hirschsohn's Test.

Process.—6 drops of reagent, made by dissolving one gram AuCl_3 in 200 cc. chloroform, were added to 5 cc. oil and well shaken.

Results Obtained.

A. Phoenix Paint Oil—Glucose Sugar Oil.—Pale-green coloration.

B. Distillery Oil.—Yellowish-brown coloration.

COMPARISON WITH RESULTS OF OTHER OBSERVERS.

Becchi.	Brullé.	Wellman.	Observer.	Reference.
Slightly dark'd		Green; blue NH_3	Hart ¹	Chem. Ztg., 17, 1522.
Faint brown	Black		De Negri ²	Chem. Ztg., 22, 961-976.

ELAÏDIN REACTION.

Poutet's Method.

1 cc. mercury was dissolved in 12 cc. cold HNO_3 of 1.42 sp. gr.

¹ Dark brown oil.

² Petroleum ether extract.

2 cc. of the freshly prepared green solution were added to 50 cc. oil contained in a wide-mouthed, stoppered bottle, the contents of the bottle violently shaken and the agitation repeated every ten minutes for two hours. The temperature during this operation ranged from 20° C. to 22° C. The oil was then allowed to stand undisturbed in a warm room.

Results Obtained.

A. Phoenix Paint Co. Oil.—2 hrs.—Orange-yellow deposit, pastry in consistency and small in amount. Orange-red, honey-like liquid above.

24 hrs.—Deposit unchanged, supernatant liquid of less viscosity and darker color.

2 wks.—Little change except in gradual darkening of liquid to reddish-brown.

B. Chicago Glucose Sugar Refining Co. Oil.—Reaction similar to that with Phoenix Paint Oil.

C. Distillery Oil.—Reaction similar to that with the Phoenix Paint Oil, except that the viscosity of the supernatant liquid was less and its color darker. After two weeks no difference could be detected in the results of the tests on the oils.

COMPARISON WITH RESULTS OF OTHER OBSERVERS.

Result of test.	Observer.	Reference.
Pasty or buttery mass	Smith	J. Soc. Chem. Ind., 11, 504.
Yellow-orange color; partial solidification.	Shuttleworth	Pharm. J., 16, 1095.
Yellow-orange; no solidification.	Dulière	J. Pharm. (1897), 217.
Much olein; bet. olive and cotton-seed oils.	Lloyd	Chem. Centrbl., 59, 1193.

DETERMINATION OF RISE IN TEMPERATURE WITH H_2SO_4 .

Maumené Test, Archbutt's Method.

50 grams oil were weighed into a beaker and brought to exactly the same temperature as the H_2SO_4 to be used for the test. This temperature should not be far from 20° C. but the exact point is immaterial, provided it is uniform for both oil and acid. The beaker of oil was then placed in an asbestos nest and 10 cc. H_2SO_4 , of 1.842 sp. gr. by Westphal balance, rapidly delivered into oil from a pipette. During the delivery of the

acid and thereafter, the mixture of oil and acid was vigorously stirred with a thermometer until the temperature began to fall. A record was taken of the highest temperature shown by the thermometer. The rise in temperature for 50 grams distilled water, under exactly the same conditions, was also noted. Care was observed that the examination of all the specimens of oil should be conducted with a uniform initial temperature.

Specific Temperature.

Rise in temperature of oil as compared with that of water, reckoned as 100.

RESULTS OBTAINED.

	Initial temp.	Av. rise in temp.	Specific temp.
Distilled water.....	23° C.	42° C.	100
Phoenix paint oil	23° C.	75° C.	178.6
Glucose sugar oil	23° C.	74° C.	176.2

COMPARISON WITH RESULTS OF OTHER OBSERVERS.

Maumené figure.	Observer.	Reference.
56° C.	Spüller	Dingl., 264, 626.
60.5° C.	Hart	Chem. Ztg., 17, 1522.
79° C.	Jean ¹	J. Soc. Chem. Ind., 11, 504.
84° C.-89° C.	De Negri and Fabris ¹	Ztschr. anal. Chem., 33, 547-72.
89° C.	Mills ²	J. Soc. Chem. Ind., 11, 504-5.

DETERMINATION OF HEAT OF BROMINATION.

Process of Hehner and Mitchell.

One gram of oil was carefully weighed into short test-tube having one-inch bore. This tube was then suspended in a beaker by means of a large cork and the beaker placed in an asbestos case. A double protection was thus afforded against the radiation of heat.

10 cc. of chloroform were next added to the oil and a delicate thermometer introduced into the solution, in such a manner that its bulb rested at about the center of the liquid. One cc. of bromine was then added to the dissolved oil and the rise in temperature noted. Great care was taken that the oil, chloroform and bromine should all be of the same temperature before beginning the operation.

¹ Obtained by use of Jean's Thermelaeometer.

² 15 grams oil + 5 cc. H₂SO₄.

Six readings were taken for each sample. Glacial acetic acid was used as the diluent, instead of chloroform, in the case of the mixed insoluble fatty acids. The calculated iodine value was found by multiplying the bromine thermal value by 5.5.

RESULTS OBTAINED.

	Br. Therm. Val.	Hübl No.	Calc. I. No.
Phoenix oil.....	21.9° C.	119.74	120.45
Glucose sugar oil.....	21.8° C.	118.62	119.90

COMPARISON WITH RESULTS OF OTHER OBSERVERS.

Br. Therm. Val.	Calc. I. No.	Observer.	Reference.
21.5	118.25	Hehner	J. Soc. Chem. Ind., 16, 87.

DETERMINATION OF SOLUBILITY IN GLACIAL ACETIC ACID.

Valenta's Process.

Three cc. glacial acetic acid were added to 3 cc. oil in a wide-mouthed test-tube. The mixture was then gently warmed until the oil was completely dissolved, a thermometer inserted and the oil allowed to gradually cool, with continual stirring. The temperature at which permanent turbidity first appeared was recorded.

Results Obtained.

- A. Phoenix Paint Co. Oil.*—74° C., average of six tests.
B. Chicago Glucose Sugar Refining Co. Oil.—80° C., average of five tests.
C. Distillery Oil.—44° C., average of five tests.

COMPARISON WITH RESULTS OF OTHER OBSERVERS.

Turbidity temp.	Observer.	Reference.
65° C.	De Negri ¹	Chem. Ztg., 22, 961-976.

DETERMINATION OF OXYGEN-ABSORPTION.

Livache's Test.

Finely divided lead powder was obtained by precipitating metallic lead from $(\text{CH}_3\text{COO})_2\text{Pb}$ by means of zinc, washing the precipitate rapidly with water, alcohol, and ether, in the order named, and drying it in a desiccator.

Approximately one gram of lead powder, prepared as above, was spread in a thin layer on a large watch-glass and a few

¹ Petroleum ether extract.

drops of oil added by means of a pipette, care being taken to keep the drops of oil separate. The amount of oil taken was accurately determined and was not allowed to exceed 0.6 gram. The watch-glass was then exposed to light but protected from dust, and allowed to remain, at the ordinary temperature, until it ceased to gain in weight.

The total gain in weight and the time required for this gain, were both taken into account. All samples tested were examined at same time and under identical conditions.

RESULTS OBTAINED.

A. PHOENIX PAINT CO. OIL.

Wt. oil. Gram.	Total gain. Gram.	Time required. Days.	Gain. Per cent.
0.5193	0.0310	7	5.97

B. CHICAGO GLUCOSE SUGAR REFINING CO. OIL.

0.3313	0.0172	10	5.19
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PREPARATION OF INSOLUBLE FATTY ACIDS.

The mixed insoluble fatty acids were prepared, in considerable bulk, for analysis, by a process analogous to that used in determining the Reichert value, except that no attempt was made to have the work quantitative. The oil was saponified by aqueous KOH in considerable excess, the soap decomposed by dilute H_2SO_4 , and the resultant liquid heated until the oily layer of liberated fatty acids became clear and transparent. The whole mass was then transferred to a separating funnel, the aqueous layer drawn off and the fatty acids washed with boiling water until the wash-water was neutral to litmus.

The insoluble acids were then subjected to a steam distillation at the ordinary pressure and the residue washed with boiling water, filtered through a dry filter and dried at $100^\circ C$. Considerable difficulty was found in obtaining uniform samples for analysis, owing to the sharp separation of the acids into a solid and a liquid portion.

DETERMINATION OF MELTING-POINT.

*Method of La Sueur and Crossley.*¹

A small amount of the mixed insoluble fatty acids was intro-

¹ J. Soc. Chem. Ind., Nov. 30, 1898.

duced into a thin-walled test-tube of small bore and the test-tube attached to a delicate thermometer by means of rubber bands. A capillary tube, open at both ends and having an internal diameter of not more than three-fourths of a millimeter, was then introduced into the test-tube, in such a manner that one end of the capillary dipped below the surface of the solidified fat. The whole apparatus was then introduced into a beaker filled with cold water and the temperature of the water very gradually raised.

Melting-point.

Temperature at which liquid rises in the capillary tube.

DETERMINATION OF SAPONIFICATION VALUE.

A. Method by Titration.

5-10 grams of mixed insoluble fatty acids were weighed into a small flask, dissolved in carefully neutralized alcohol by heating on the water-bath and titrated, while still warm, with n/KOH .

B. Koettstorfer Method.

As a confirmatory test, the saponification value was also found by the ordinary Koettstorfer method used in the case of the original oil.

Mean Molecular Weight of Fatty Acids.

This value, also known as the "Mean Combining Weight," is equal to the number of milligrams of fatty acids neutralized by 1 cc. of n/KOH .

The "Saponification Equivalent" of fatty acids is identical with their mean molecular weight. This is not true in the case of neutral oils.

Determination of Constants.¹

A. SPECIFIC GRAVITY.

	Wt. acids at 100° C. Gram.	Wt. water at 15.5° C. Gram.	Sp. gr. acids at 100°.
I.	0.3250	0.3811	0.8528
II.	0.6780	0.7947	0.8530
Average			0.8529

¹ The fatty acids examined were prepared from the Glucose Sugar Refining Company's oil.

B. MELTING-POINT.

22.4° C..... Average of four tests.

C. IODINE ABSORPTION.

	Wt. mixed acids. Gram.	Vol. hypo. ¹ cc.	Wt. iodine. Gram.	Iodine abs. Per cent.
I.....	0.2794	26.4	0.33528	120.0
II.....	0.2523	24.4	0.30988	122.8
III.....	0.1752	16.7	0.21209	121.1
IV.....	0.3156	29.8	0.37846	119.9
Average Hübl figure.....				120.98

D. HEAT OF BROMINATION.

	Br. Therm. Val.	Hübl No.	Calc. I. No.
Fatty acids.....	21.6° C.	120.98	118.80

E. SAPONIFICATION VALUE.

	Wt. acids. Grams.	Vol. n /KOH by titr. cc.	Saponifi- cation val.	Mean combin. wt.
I.....	7.0294	25.0	199.52	281.17
II.....	5.6080	19.7	197.06	284.70
		By Koett.		
III.....	1.8415	6.5	198.02	283.30
IV.....	1.4302	5.15	202.01	277.71
Average.....			199.15	281.72

COMPARISON WITH RESULTS OF OTHER OBSERVERS.

B. MELTING-POINT.

Melting-point acids.	Observer.	Reference.
10.5° C.-12.2° C.	Hoppe-Seyler ²	Bull. Soc. Chim. (1866), [2] 6, 342.
16° C.-18° C.	Dulière	J. Pharm. (1897), 217.
18° C.-20° C.	De Negri and Fabris	Ztschr. anal. Chem., 33, 547.
20° C.	Jean	J. Chem. Soc. Ind., 11, 504.
39.5° C.	De Negri ³	Chem. Ztg., 22, 961-976.

C. IODINE ABSORPTION. ²⁸

Hübl No. acids.	Observer.	Reference.
113-115	De Negri and Fabris	Ztschr. anal. Chem. 33, 547.
123.27	De Negri ³	Chem. Ztg., 22, 961.
125	Spüller ²	Dingl., 264, 626.
126.4	Hopkins	J. Am. Chem. Soc., Dec., 1898.

E. SAPONIFICATION VALUE.

Sapon. value.	Mean mol. wt.	Observer.	Reference.
198.4	282.76	Hart	Chem. Ztg., 6, 1522.

¹ 24.8 grams per liter.² Ether extract.³ Petroleum ether extract.

A METHOD FOR THE IMPROVEMENT OF MAIZE OIL BY THE USE
OF STEAM.

Introductory Statement.—As developed in the course of the preceding analysis, the oil of maize has several objectionable properties, which tend to injure it commercially for uses for which it would be otherwise well-fitted. Among the most important of these properties are the following:

1. The pronounced grain-like odor and the acrid after-taste of the oil. These qualities unfit it for use as a salad oil.
2. The rather high percentage of free acid and the marked action which the oil has on metal. These qualities lessen its value as a lubricating oil.
3. The slowness with which the oil absorbs oxygen, even under the influence of dryers. This militates against its use as a paint oil or, at best, permits it to be used only in a cheap grade of paint.

It was with the idea of improving maize oil in one or more of these directions that it was subjected to a prolonged treatment with steam. A somewhat extended study of the work of other observers on this oil, fails to reveal any attempt to modify the oil in this way. It therefore seems a fair conclusion, to claim that the application of steam distillation to maize oil, for the purpose of improving it commercially, is a new method.

Description of Process.—A fresh sample of the oil, as expressed from the germ by hydraulic pressure,¹ was subjected to a brisk steam distillation for about eight hours. Just enough heat was used under the oil to prevent condensation and the steam was used at the ordinary pressure.

The odor of the volatile oil was very marked, particularly at the beginning of the operation, and a portion of this oil was afterward recovered from the distillate.

Considerable dissociation of the lower esters took place and large quantities of a solid fatty acid, volatile to some extent in steam but insoluble in water, collected in both the retort and the receiver.

At the close of the operation, the liquid in the distilling flask was transferred to a separating funnel, the aqueous layer drawn off and the oil washed repeatedly with boiling water. The oil

¹ From the Chicago Glucose Sugar Refining Co.

was then allowed to stand for sometime in the separating funnel in order that the water might drain out. After as complete separation as could be effected in this way, CaCl_2 was added to the oil and the drying completed. The oil was then filtered through a dry filter until bright and clear and the constants determined in the usual manner.

Determination of Constants.

A. SPECIFIC GRAVITY.

	Wt. water 15.5° C. Grams.	Wt. oil 15.5° C. Grams.	Wt. oil 100° C. Grams.	Sp. gr. 15.5° C.	Sp. gr. 100° C.
I	1.7733	1.6371	1.5477	0.9232	0.8729
II	1.1522	1.0642	1.0062	0.9236	0.8733
Average				0.9234	0.8731

B. FREE ACID.

	Wt. oil. Grams.	Vol. $n/_{10}$ KOH. cc.	Acid value.	Free acid. Per cent.	Degrees acidity.
I	9.4952	2.9	1.71	0.861	3.05
II	5.3112	1.6	1.69	0.849	3.01
Average			1.70	0.855	3.03

C. SAPONIFICATION VALUE.

	Wt. oil. Grams.	Vol. n/HCl . cc.	Koettstorfer fig.	Sapon. equiv.	Ether value.
I	2.7542	9.24	188.21	298.07	186.51
II	2.2459	7.64	190.84	293.96	189.14
Average			189.53	296.02	187.83

D. IODINE ABSORPTION.

	Wt. oil. Gram.	Vol. hypo. cc.	Wt. iodine. Gram.	Iodine abs. Per cent.
I	0.2624	23.8	0.296334	112.93
II	0.2522	22.9	0.285124	113.05
III	0.2743	24.9	0.310029	113.03
IV	0.2434	21.9	0.272676	112.03
Average Hübl figure				112.76

E. ACETYL VALUE.

	Wt. oil. Grams.	Vol. n/HCl . cc.	Koettstorfer fig.	Vol. $n/_{10}$ KOH. cc.	Acetyl figure.
I	2.0468	8.0	219.27	4.5	12.3
II	2.3814	9.2	216.73	5.1	12.0
Average			218.00	...	12.15

F. MAUMENÉ FIGURE.

Initial temp. 23° C.	Av. rise in temp. 77° C.	Specific temp. 183.3
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G. BROMINE THERMAL VALUE.

Br. thermal val.	Hübl No.	Calc. iodine No.
20.4° C.	112.76	112.20

H. VISCOSITY.

Tempera- ture.	Av. time of flow. Seconds.	Viscosity. Water.	Viscosity. Rape oil.
20° C.	310.2	10.70	76.50

K. INDEX OF REFRACTION.

	Temperature.	A.	T.
I	15° C.	1.4765	35.7
II	15° C.	1.4766	35.8
III.....	15° C.	1.4766	35.8
Average for 15° C.....		1.4766	35.8

L. SOLUBILITY IN GLACIAL ACETIC ACID.

Valenta value = 60° C.....Average of four tests.

M. SILVER NITRATE REDUCTION TESTS.

Becchi's testDark brown coloration.

Brullé's test.....Black coloration.

N. OXYGEN ABSORPTION BY LIVACHE TEST.

Wt. oil. Gram.	Total gain. Gram.	Time required. Days.	Gain. Per cent.
0.3005	0.0105	4	3.50

Effect of Treatment as Shown by Comparison.—In instituting this comparison, reference is made only to the sample from the Glucose Sugar Refining Co., since it was this oil which was chosen as the subject for experiment.

A tabulation of the constants of the steamed oil, together with those of all other samples examined, will be given in the summary of this dissertation. but the main points of difference between the treated and untreated oils will be noted here.

1. The steamed oil is notably lighter in color than that not so treated.

2. The odor and taste of the volatile oil are entirely lacking in the modified oil, which is absolutely neutral in these respects.

3. The specific gravity and acetyl value are somewhat increased, but not to the extent which might naturally be expected.

4. The percentage of free acid is notably decreased, this constituting a marked improvement in the oil.

5. The solubility of the oil in alcohol and in acetic acid is noticeably increased.

6. The iodine absorption, bromine thermal value and absolute amount of oxygen absorption are all decreased, but the total time required for drying is much lessened. The drying qualities of the oil are, therefore, on the whole, considerably improved.

Further investigation is needed as to length of treatment, effect of age on the modified oil and several other points, but enough work has been done to show that, by the process in question, maize oil is much improved for use as a salad oil, a lubricant, or even as a paint-oil.

PART III—SUMMARY.

DESCRIPTION OF SAMPLES.

I. Chicago Glucose Sugar Refining Co. Oil.

This oil is a freshly made, pale yellow sample of maize oil, as expressed from the germ by hydraulic pressure, and illustrates the ordinary commercial product.

II. Phoenix Paint Co. Oil.

This specimen is a ten-year-old sample of maize oil, which was also obtained by expression from the germ, and which has been preserved from the action of light and air. It serves to illustrate the changes in the oil which are due to age.

III. Distillery Oil.

This oil is a golden-brown sample of maize oil from the mash of a distillery and is approximately fifteen years old. As will be seen from an examination of its constants, it is practically a distinct oil from that prepared by hydraulic pressure. For this reason, its examination was abandoned after the determination of its more important constants.

IV. Insoluble Fatty Acids.

This sample was prepared from the Glucose Sugar Co. oil by a method given in detail on page 30 of this dissertation.

V. Steamed Maize Oil.

A full description of this modification of maize oil, together with its mode of preparation, will be found on pages 33-36.

TABLE OF PHYSICAL CONSTANTS.

	Glucose oil.	Phoenix oil.	Distillery oil.	Insol. Fat acids.	Steamed oil.
Specific gravity	0.9213	0.9213	0.9255		0.9234
Specific gravity	0.8711	0.8716	0.8756	0.8529	0.8731
Viscosity (water)	10.27	9.79			10.70
Viscosity (Rape)	73.89	70.42			76.50
Index of Refrac.	1.4766	1.4767			1.4766
Index of Refrac.		1.4761	1.4765		
Melting-point				22.4° C.	

TABLE OF CHEMICAL CONSTANTS—QUANTITATIVE.

	Glucose oil.	Phoenix oil.	Distillery oil.	Insol. Fat acids.	Steamed oil.
Per cent. ash.....		0.065	0.0655		
Acid value	2.25	3.70	20.65		1.70
Per cent. free acid ..	1.128	1.851	10.386		0.855
Degrees acidity.....	4.00	6.59	36.83		3.03
Per cent. iodine abs.	118.62	119.74	113.27	120.98	112.76
Koettstorfer fig....	192.64	192.65	191.78	199.15	189.53
Saponifica'nequiv..	291.22	291.21	292.55	281.72	296.02
Ether value	190.39	188.95	171.13		187.83
Hegner value.....	92.23	92.79	88.21		
Reichert figure...	4.2	4.3	9.9		
Wt. KOH per 100 grams oil.....	0.94248 g	0.96492 g	2.22156 g		
Koettstorfer of acet. oil.....	211.9	211.5			218.0
Acetyl value.....	11.49	11.12			12.15
Per cent. glycerol ..	10.35	10.545			
Percent. unsap. mat.	1.43	1.39			

TABLE OF CHEMICAL CONSTANTS—MISCELLANEOUS.

	Glucose oil.	Phoenix oil.	Distillery oil.	Insol. fat acids.	Steamed oil.
Heydenreich— H_2SO_4	Mahogany red to dark red brown	Golden brown; violet after 24 hrs.	Mahog. to claret	...	
Carbon disulphide— H_2SO_4			Claret; violet in 24 hrs.		
Hauchecorne— HNO_3	Orange yellow		Mahogany		
Massie— HNO_3	Mahogany red		Dark red brown		
Glassner— HNO_3	Pale yellow solid		Orange yellow		
Becchi— $AgNO_3$	Dark brown		Dark brown		Dark brown
Brullé— $AgNO_3$	Black		Black		Black
Wellman—Phos. mol. acid	Dark green changed to bright blue		Light green and blue		
Renard— $SnBr_4$	Brown-violet		Brown-violet		
Hirschsohn— $AuCl_3$	Pale green		Yellow brown		
Elaidin test	Orange yellow deposit; red liquid		Orange solid; dark red liq'd		
	74° C.	75° C.			77° C.
Maumené—Rise in temp.	176.2	178.6			183.3
Maumené—Specific temp.	21.8° C.	21.9° C.		21.6° C.	20.4° C.
Bromine thermal value	80° C.	74° C.	65° C.		60° C.
Valenta's test					3.50 in 4 days
Livache test—Per cent. gain	5.19 in 10 days	5.97 in 7 days.			

MATHEMATICAL CALCULATIONS.

By means of the following calculations, an attempt has been made to show that the results of the preceding analysis are consistent and that, at most, the difference between the observed and the calculated amounts is well within the bounds of experimental error.

The mean molecular weight of each class of fatty acids and their relative amounts, are also here derived. In this way an idea is gained as to the constitution of the oil, which may serve as the basis of further investigation.

I. Determination of Glycerine.

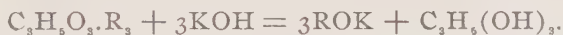
Assume R = radicle of fatty acid = *e. g.* $C_{18}H_{36}O$ = radicle of $C_{18}H_{36}O_2$.

$\therefore C_3H_5O_3 \cdot R_3 = (ROH)_3 \cdot C_3H_5 =$ triglyceride.

Assume M = molecular weight of ROH .

$\therefore 3M + 38 =$ molecular weight of triglyceride.

Reaction for Saponification.—



Assume E = ether value of glucose oil.

$\therefore$ Per cent. glycerine = $\frac{E \times \text{mol. wt. glycerine} \times 100}{3 \times \text{mol. wt. KOH} \times 1000} = 10.41.$

Per cent. glycerine actually recovered = 10.35.

II. Determination of Total Fatty Acids.

A neutral glyceride may be considered as made up of three molecules of a fatty acid and one group C_3H_5 . The component parts of any oil are, therefore,

1. Total fatty acids.
2. $\frac{38}{92}$ of total amount of glycerine.
3. Unsaponifiable matter.

$\therefore$ 100 grams maize oil = F grams total acid +

$$\overbrace{\frac{38}{92} \times 10.41 \text{ grams}}^{\text{Calc. glyc.}} + \overbrace{1.43 \text{ grams}}^{\text{Unsap. mat.}}$$

$\therefore$ Per cent. total fatty acids = 94.27.

III. Determination of Volatile Acids.

Observed Hehner value = 92.23

$$\therefore \text{Per cent. volatile acids} = 94.27 - 92.23 = 2.04.$$

IV. Molecular Weight of Total Acids.

Observed saponification equivalent = 291.22.

$$\therefore \text{Mol. wt. total acids} = 291.22 \times 0.9427 = 274.533.$$

V. Molecular Weight of Volatile Acids.

$$\frac{\text{Per cent. total acid}}{\text{Mean mol. wt. total acid}} = \frac{\text{Per cent. insol. acid}}{\text{Mean mol. wt. insol. acid}} + \frac{\text{Per cent. vol. acid}}{\text{Mean mol. wt. vol. acid}}$$

$$\therefore \frac{94.27}{274.533} = \frac{92.23}{281.72} + \frac{2.04}{\text{Mol. wt. vol. acids}}$$

$$\therefore \text{Mean mol. wt. volatile acids} = 127.5.$$

VI. Determination of Reichert Figure.

Reaction between KOH and fatty acid is of the form



$\therefore$ Wt. KOH equivalent to volatile acids from 2.5 grams oil is

$$\frac{5.61 \times 0.0204 \times 2.5}{127.5} = 0.02244 \text{ gram.}$$

$$\therefore \text{Calculated Reichert figure} = 4.$$

$$\text{Observed Reichert figure} = 4.2.$$

Consistency of Observed Constants.

1. Since the calculated percentage of glycerine is dependent on both the acid value and the Koettstorfer figure, the consistency of these values with each other and with the observed glycerine content may be considered proved.

2. The close agreement between the calculated and observed Reichert figures further demonstrates the correctness of the Hehner value, the Koettstorfer value, the molecular weight of the insoluble acids and the percentage of unsaponifiable matter.

3. The harmony between the bromine thermal values and the Hübl figures is evidence of the correctness of these constants as

observed. The consistency of the following observed constants may therefore be assumed to be established.

1. The acid value.
2. The Koettstorfer figure.
3. The percentage of glycerine.
4. The Hohner value.
5. The Reichert figure.
6. The molecular weight of the insoluble fatty acids.
7. The percentage of unsaponifiable matter.
8. The bromine thermal value.
9. The Hübl figure.

PERCENTAGE COMPOSITION OF MAIZE OIL.

Neutral glycerides.....	97.4418
Free acid	1.1280
Unsaponifiable matter	1.4300
	99.9998

CLASSIFICATION OF FATTY ACIDS OF MAIZE OIL.

	Per cent.	Mean mol. wt.
Total acids.....	94.27	274.533
Insoluble acids	92.23	281.720
Volatile acids.....	2.04	127.500

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BIOGRAPHICAL.

Harriet Winfield attended the Jersey City High School for four years and Wellesley College for four years. At Columbia University, New York, she studied under the Faculty of Pure Science during the years 1895-6, '96-7, '97-8, and '98-9.

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